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ORIGINAL ARTICLE

Measurement of yield stress for concentrated suspensions using a plate device

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Abstract Slump has often been correlated with the yield stress of concrete as defined by the Bingham model. The discussion is still open as to what the yield stress value actually is and how to measure the yield stress of a suspension in general and for a cementitious material in particular. A plate device is a recent development in the measurement of yield stress of suspensions that allows for testing at shear rates far below most rotational rheometers. This paper presents the plate device and the modifications made so that it can be used with suspensions such as bentonite or titanium dioxide (TiO_2) in aqueous solution, as well as high concentration suspensions such as portland cement paste. A systematic analysis of the experimental results is presented with a critical discussion on the potential use of this device. The results indicate that the yield stresses of the suspensions determined by the plate device were generally lower than those determined by the parallel-plate rheometer. It appears that the pattern of stress growth curve and method of yield stress calculation in plate device experiments are affected by the suspension type.

Keywords Cement paste · Plate device · Suspension rheology · Yield stress · Workability

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1 Introduction

The workability of fresh concrete is an important property that affects the consolidation, mechanical properties, and durability of concrete. From a rheological perspective, the flow behavior of mortar and concrete is usually described by the Bingham model with two parameters—yield stress and plastic viscosity.

According to Hackley and Ferraris [1], the physical definition of the yield stress is the stress needed to initiate movement; therefore, it should be measured by slowly increasing the shear stress until movement occurs. This direct measurement is not easy to implement in most rotational rheometers because it requires that the shear stress is very well controlled. Most rheometers cannot control the stress with steps small enough to detect the yield stress.

Most commonly used rotational rheometers are coaxial or parallel plate types (ACI 238-1R [2]). One difficulty in measuring the yield stress and plastic viscosity of suspensions using such equipment is the wall–sample interaction, which may result in slip effects. According to Zhu et al. [3] and Zhu and De Kee [4], wall depletion effects with multi-phase systems are associated with the displacement of the dispersed phase(s) away from solid boundaries, leaving a low-viscosity, particle-depleted layer near the wall. This phenomenon is due to surface, hydrodynamic, viscoelastic, chemical, and gravitational forces acting on the dispersed phase immediately adjacent to solid boundaries. The probability of wall slip increases when dealing with smooth walls, relatively small gaps, low flow rates [5], and suspensions of large or flocculated particles. The low viscosity of the liquid near the wall leads to lower measured shear stress at a fixed shear rate, and therefore the viscosity and the yield stress are under-estimated [5]. A common way to avoid such complications is to roughen the surface of the walls in order to increase the sample-wall friction.

To overcome such complications, Zhu et al. [3, 6] and Zhu and De Kee [4] developed a plate device and method to measure the yield stress of suspensions using a controlled low shear rate. The novelty of the method is that the plate is slotted, and designed to create suspension/suspension shearing and not shearing between a "wall" and a suspension. This would hopefully eliminate the wall effect, and lead to a measurement of the true yield stress of a suspension. According to Zhu et al. [3], the method is particularly useful for measuring low-concentration suspensions with very low yield stresses. This method has been further developed by Picandet et al. [7] for concentrated suspensions such as cement pastes. However, many issues regarding the use of the device for yield stress measurement of concentrated suspensions remain.

Another difficulty is that most rotational rheometers have difficulties to apply a low shear rate, and they are expensive. The plate device overcomes these limitations by allowing to select a linear movement that can control a low shear rate a lot easier and cheaper.

In this paper, the device built by Picandet et al. [7] at the National Institute of Standard and Technology (NIST) from the design of Zhu et al. [3] and Zhu and De Kee [4] is exploited to measure cement paste, bentonite and titanium dioxide (TiO_2) suspensions. A systematic analysis of the experimental results is presented with a critical discussion on the potential use of this device.

2 Background

The flow of a material such as cement paste (cement particles suspended in water) is difficult to measure due to the presence of particles. The overall behavior is non-Newtonian. The main characteristic of such a material is that it behaves as an elastic solid until it starts flowing. The shear stress at which the material starts flowing is called the yield stress. The yield stress is not easy to determine because the material needs to be sheared at very slow rate (zero shear rate limit) to capture it. Zhu et al. [3] have shown that the yield stress depends on the static shear rate used until the shear rate is below a certain value as measured using the plate device. In the case of TiO_2 suspensions, they found that the speed should be below 0.1 mm/min (1.7×10^{-3} mm/s) for their plate device. As very few instruments can shear at such a low rate, it is not known whether this upper limit is material dependent. Obviously, the practical question arises as to whether knowing the true yield stress is necessary for engineering purposes as a material like concrete is seldom sheared at such low shear rates in a construction site.

Most commercially available rotational rheometers cannot achieve a low enough rotational speed, therefore usually the yield stress is measured either by extrapolation to zero shear rate using some model such as Bingham or by stress growth (Fig. 1) at a relative high speed in the order of 10 mm/s at the outer edge of a parallel plate.

Yield stress may be calculated based on the net force at Point A or Point B as shown in Fig. 1. Some researchers [8, 9, 10] suggest that the shear stress calculated from the force at Point B corresponds to complete break down of the fluid structure and hence is a measure of the yield stress. However, other studies [11–13] suggest that the non-linear region from Point A to B is a function of the applied shear

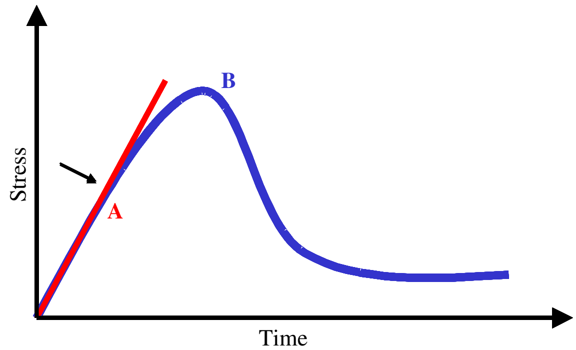


Fig. 1 Schematic of a stress growth measurement. Point A is the end of the linear portion, i.e., elastic portion, and it is the static yield stress point. Point B is the peak point associated with the dynamic yield stress and it is taken as an approximation of the true yield stress because it is easier to determine than Point A

rate and that yield actually starts at Point A where the curve deviates from linearity. The yield stress corresponding to the shear stress at Point A is often referred to as static yield stress (τ_{st}) since macroscopic flow has not occurred. The stress at the peak of the shear stress-time curve is often referred to as dynamic yield stress (τ_d), denoting the onset of viscous flow. According to Nguyen and Boger [5], these two yield stresses might be lower and upper yield stress values corresponding to the end of the elastic deformation and start of fully viscous flow, respectively. Zhu et al. [3] also showed that the shear stress at Point A does not change with shear rate applied, if the speed is low enough. On the other hand the stress at Point B will depend on the applied shear rate.

To measure the yield stress at very low shear rates, Zhu et al. [3] and Zhu and De Kee [4] developed a new device, thereafter called the plate device. The device consists of a slotted plate submerged into a test material; the plate is slowly pulled out of the suspension while measuring the required load, which comes from the resistance of the material to this motion. The relationship of the stress versus time should be linear for an elastic material.

The idea to pull or push an object through a suspension to determine the rheological parameters is not new. A review of these methods can be found in the ACI 238 [2] document. The idea is based on the falling ball rheometer and targets the measurement of viscosity by applying the Navier-Stokes law. In all previous cases, the speed was much higher than desired for a measurement of the yield stress. Another test that was very similar in some ways is the Lombardi plate [14]. The test involved a plate with a rough surface attached to a balance. The plate was pulled out of the material and the mass of material attached to the plate was measured. There was no attempt to measure the force of moving the plate out of the material. Lombardi estimated that the amount of material sticking to the plate after removal from the suspension was related to the cohesion of the material and he showed that it was correlated with the yield stress measured using a Bingham model.

The key principle of the plate device [3] is that the speed can be easily controlled using a step motor (see Sect. 3.1) and the plates can be modified to accommodate suspensions with different particle size distributions. To obtain the true yield stress the material should be sheared within the material and not between the material and an object, such as a plate. Therefore, ideally, a virtual plane of material should be moved inside the suspension and the material-material shearing stresses measured. Obviously, this is not possible in practice, so the virtual plate is approximated by a slotted plate. The slot area should be maximized while ensuring that there is no flow in the slots. The material in the slot behaves as a solid plate. Zhu et al. [3] through testing and simulation showed that the larger the slot area of the plate, the larger the measured yield stress up to a value of slot ratio β of about 0.5 ($\beta = S_{Bulk}/S$; where S_{Bulk} is the surface area of the slot and S is the surface area of the plate). Therefore, a value of β higher than 0.5 (for a TiO_2 suspension) measured the true yield stress of the material. At these values of β, the device was not affected by slip. The yield stress in a suspension was always larger than the adhesive force between the suspension and the plate.

Zhu et al. [3] compared yield stresses determined using smooth stainless steel plates and sand-blasted Ti–Al alloy plates, and found that plate material properties as well as surface roughness also influence yield stress measurement. They suggested and confirmed that the measured yield stresses of TiO_2 suspensions using two kinds of plates were very close to each other at high slot ratios while they could be significantly different at low slot ratios. This is attributed to the fact that true yield stress is independent of the plate condition as it should reflect the material-material shearing stresses as measured at high slot ratios.

Other methods could be imagined to measure yield stress, such as oscillatory techniques, stress controlled/shear rate measurement or creep recovery [1] but the discussion of their advantages and disadvantages is beyond the scope of this paper. The goal here is to examine if the method developed by Zhu et al. could be applied to materials such as cement or other pastes at similar volume concentrations. The determination of the true yield stress is essential in the description of the flow of dense suspensions such as cement pastes.

3 Plate device and test method

3.1 Equipment design

The instrument set-up from Zhu et al. [3] and Zhu and De Kee [4] was originally used for dilute suspensions. NIST built the device with some modifications to adapt it for more concentrated suspensions. This device consists of a container with the material to be tested placed on a platform that can move in the vertical direction. Figure 2 shows a schematic plan of the device. The movement of the platform is controlled by a computer through a step-motor with a minimum speed of 0.05 mm/s. The speed is checked using data acquisition. Uncertainty is estimated from the uncertainty on time and displacement, measured by the LVDT (Fig. 2), i.e. less than 0.001 mm/s. A balance is placed on a support on top of the container. The balance support cannot move and a hook below the balance suspends the plate.

Various types of plates (slotted plates and solid plates of various lengths) were used. The slotted plates (Fig. 3) were used to create suspension/suspension shearing, whereas the solid plates were used to determine shear stress as between the plate and the suspension, as well as any correction factor that was needed due to the edge effect of the plates.

The plates were laser cut from a 1.2 mm thick and 50 mm wide stainless steel sheet. Some of the plates were additionally sandblasted on both faces to roughen the surface and to minimize slippage while others were used as is. The plates were of various lengths as shown in Table 1. For the slotted plates, 11 and 14 slots with a height of 3.05 mm and a width of

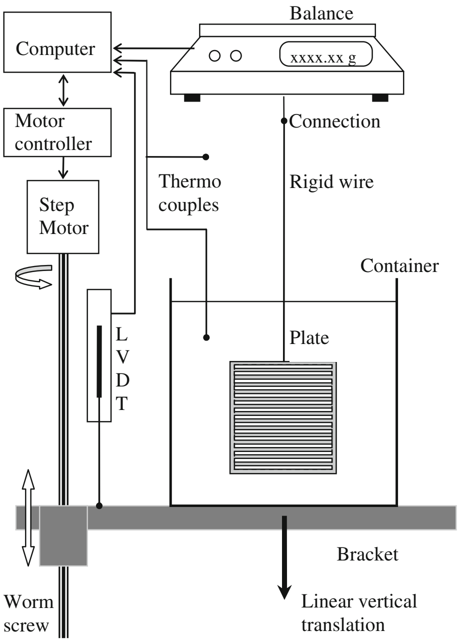


Fig. 2 Experimental set-up (not to scale) [7]

40 mm were laser cut and evenly distributed in the SL11 and SL14 plate, respectively.

It was determined [6, 7] that if the distance between the plate and the wall of the container was greater than 20 mm, the size and shape of the container had no influence on the results for the three kinds of suspensions investigated in this study.

3.2 Principles of operation

To initiate a test, the material is placed in the container and a plate is hooked to the bottom of the balance. The mass of the plate is recorded. The container is then lifted so that the plate is immersed at the desired depth. Vibration is applied to the suspension to ensure that the plate is as vertical as possible. The plate is at rest in the suspension for the first 10 min to allow for equilibrium. The platform with the container is then moved downwards at a speed of 0.05 mm/s for 5 min, for a total container displacement of 15 mm. This is followed by a rest of 5 min. Total time for each test is 20 min. The temperature of the suspension is monitored by a thermocouple during the test and recorded.

Specialized software is developed to interface with the measurements and control the step motor moving of the platform supporting the container. The software is able to record the speed and displacement of

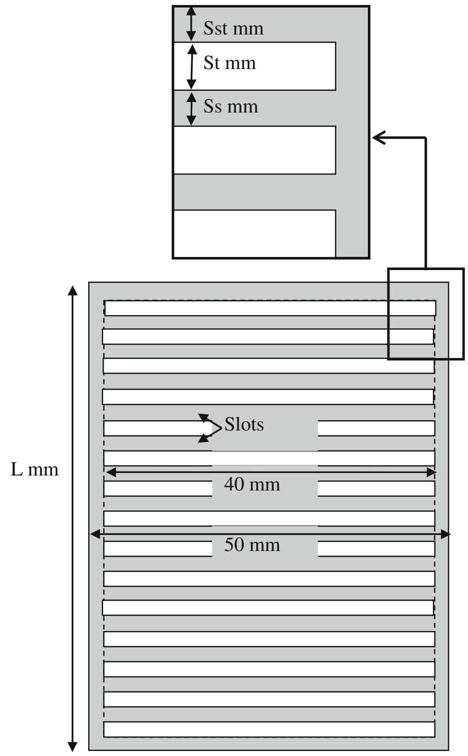


Fig. 3 Slotted plate design (not to scale). L is the length of the plate and in our case (Table 1) could be 52 mm, 75 mm or 100 mm; Sst is the thickness of the outer frame (5 mm); St is the slot width and the Ss is the steel width. St and Ss vary depending on the number of slots

Table 1 Dimension of the plates

Length of plates (mm)/slots number	Name given to plates	
	Sand blasted surface	Non sand blasted surface
52/no slot	SPS	SP
75/no slot	MPS	MP
100/no slot	LPS	LP
75/11 slots	SL11	
75/14 slots		SL14

All plates are 1.2 mm thick and 50 mm wide

the platform, the temperature of the suspension, and the output of the balance.

When the plate is immersed in the material and the container is slowly moved downwards, the material in the container exerts a force on the plate. The plate in turn exerts a force on the balance, and the resulting force and corresponding displacement are recorded by a computer in real time. The movement of the platform is measured by a linear variable differential transformer (LVDT) and controlled by a computer through a step motor.

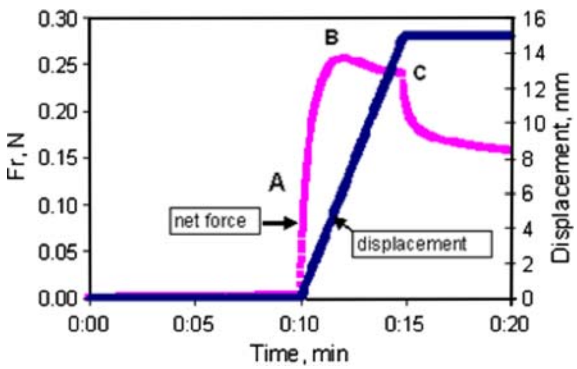


Fig. 4 Schematic strain ramp applied and typical force response obtained (bentonite suspension B1, tested with plate MPS)

A typical force and displacement versus time is shown in Fig. 4. After 10 min of rest, the measured force increases almost linearly with time, confirming the elastic nature of the material before yielding. At Point A (Fig. 4), the slope of the curve begins to decrease, and the force-time curve deviates from linearity. Just before the stress reaches its maximum, the slope of the curve decreases significantly, and the non-linear behavior between Points A and B is likely associated with structural rearrangement [3]. At the maximum stress, the structure is destroyed and the suspension flows. The region between Points B and C reflects the thixotropic nature of the suspension. If the strain is stopped at Point C, a stress relaxation is recorded. The remaining stress recorded at the end of the test is associated with a residual stress, which cannot be used to compute a reliable value of yield stress because of the thixotropic nature of these kinds of suspensions.

Zhu et al. [3] have shown, using a plate device, that the maximum stress depends on the strain rate, but that at a sufficiently low speed, around 0.01 mm/s for TiO_2 suspensions, the yield stress ceases to be a function of platform speed. However, the rheological properties of cement paste samples change with time due to the hydration kinetics of cement paste. To consider constant properties during testing, the platform speed has been selected to be 0.05 mm/s. A lower speed would not allow one to clearly observe a maximum stress as shown in Fig. 4 because the test will be too long to ensure that hydration could be neglected.

From the force recorded when the plate moves in a suspension, the shear stress τ can be calculated using Eq. 1, and shear stress-time curves can be plotted.

$$\tau = F_r/S = (F - F_i)/2WL \tag{1}$$

where F_r is measured net force, S is surface area of the plate, F is measured force using balance, F_i is initial force (= gravitational force due to plate and wire mass − the buoyant force in suspension), L is length of the plate, and W is width of the plate

3.3 Calculation of yield stress from the plate device tests

3.3.1 Correcting the edge effect

Smooth plane plates of varying lengths were used to determine the drag forces exerted on the plate edges and to evaluate τ_S, the stress from the steel surface. When the plate moves upward, its upper edge displaces the suspension and there are drag forces exerted on the lower edge F_{el} and upper edge F_{eu} of the plate (see Fig. 5).

$$F_r = F_{eu} + F_{el} + 2WL\tau_S \tag{2}$$

where F_r is measured net force at the yield point, F_{el} is force exerted at the lower edge, F_{eu} is force exerted at the upper edge, L is length of the plate, W is plate width, and τ_S is yield stress associated with the surface $2WL$.

W, τ_S and the sum of the drag forces $F_D = F_{eu} + F_{el}$ is constant for a given mix. The series of stainless steel plates with different lengths but same edge dimension, geometry and surface conditions (Fig. 5) was used to correct for the edge effect by plotting F_r vs. L which generated a straight line with intercept F_D and with a slope related to τ_S (Fig. 6).

3.3.2 Slotted plates

To analyse the slippage effect, a series of parallel and horizontal slots were machined into a medium sized plate (Fig. 3). It was assumed that when the slotted plate moves in the material, the suspension in a slot remains static relative to the plate. Zhu et al. [6] have shown that if the ratio of the height of a slot over the thickness of the plate is smaller than three, the suspension filling the slots can be considered static, with no secondary flow and with shearing occurring only at the edge of the slots.

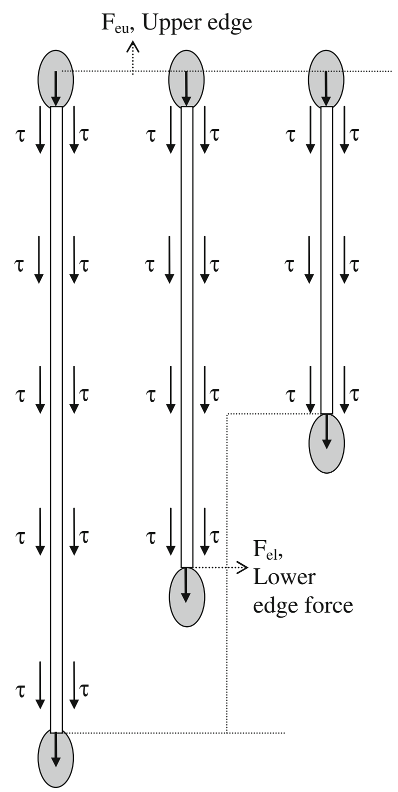


Fig. 5 Exerted forces on the plates

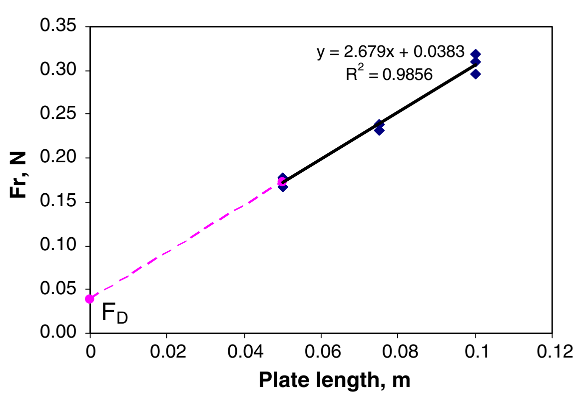


Fig. 6 Typical graph of exerted forces on the plates of various lengths (bentonite suspension B1, tested with smooth plates)

Using the slotted plate, the suspension yield stress τ_B is measured at the interface of the suspension inside the slots and the adjacent bulk. The mean stress exerted on the plate is the combination of the yield stress τ_B over an area $S_{Bulk} = \beta S$ and τ_S over an area $(1 - \beta)S$. τ_S is assumed to be lower than τ_B due to slip. The mean shear stress τ exerted on the plate is a function of the area ratio $\beta = S_{Bulk}/S$, τ_S, and the parameter to be evaluated, τ_B:

$$\tau = [\beta\tau_B + (1-\beta)\tau_S] \quad (3)$$

The force F_r exerted on the plate is:

$$F_r = 2WL[\beta\tau_B + (1-\beta)\tau_S] + F_D \quad (4)$$

The drag force F_D depends on the slope of the sample as well as on the operating conditions such as plate speed and test temperature, but it is not a function of the slot ratio β since the suspension in the slots moves with the plate. The ideal situation would refer to a virtual sample plate moving through the sample or a plate with one large slot but the material would need to be in the slot and not move.

Two slotted plates (Table 1) were used for the actual measurements: (1) a plate with a sand blasted surface with $\beta = 0.358$ (SL11) and (2) a plate with a smooth surface with $\beta = 0.455$ (SL14). The measurements with the two plates allow for the extrapolation of the contribution of the slip effect and the deduction of the yield stress τ_B of the suspension [6]. The slotted plates were designed such that the ratio of the height of the slots over the thickness of the plate was less than three, and the slot height was at least 100 times larger than the particle size in the suspensions [3]. These conditions were met to strengthen the assumption that the suspension in the slots was static with no secondary flow and the shear occurred only at the edge of the slots (material in the slot shearing against the material in the bulk). The method also assumes that the structure of the suspension in the slots is identical to that of the bulk suspension, and that the stresses τ_S and τ_B are independent.

4 Experimental study

4.1 Materials used and mixtures

An ASTM Type I normal portland cement, three bentonites, and a commercial TiO_2 pigment,[1,2] were used in this research. Table 2 summarizes the mixtures. Information on how the mixtures were prepared

Table 2 Summary of the mixture used

Solid particles	Water/solid ratio (by mass)	Additives and dosage (% of solids by mass)	Volume concentration of solids (%)
Cement	0.33	Retarder (0.19%)	48
Bentonite B1	9.0[a]	–	4.0
Bentonite B2	9.3	–	3.1
Bentonite B3	9.3	–	3.1
TiO_2	1.0	–	20

[a] The mass concentration of bentonite B1 suspension was determined by averaging three tests as the moisture content of B1 was not measured prior to preparing the suspension

and on the composition of the various materials are given below. More details can be found in [15].

4.1.1 Cement paste

The ASTM Type I normal portland cement had a specific surface area of 541 m^2/kg calculated from the particle size distribution as measured by laser diffraction (Fig. 7).

The cement paste suspension had a water/cement (w/c) ratio of 0.33 by mass. This 48% concentration by volume in water can be considered to be a high concentration suspension. A retarder admixture was added to allow for a longer time to conduct the measurements.

Cement paste was mixed with a Hobart[2] mixer for about 4 min initially, and then mixed intermittently every 5 min until about 34 min from the time water was in contact with the cement. A retarding admixture was added in the mixing water to delay the onset of cement hydration, so that workability of the cement paste remained almost constant during a period of about 3.5 h. After mixing, the paste was transferred to a plastic container with a diameter of approximately 115 mm.

The yield stress was also determined via a parallel plate rheometer using a stress growth method (Point B of the stress versus time at constant shear rate) [16] up to about 7 h, and the results indicated that the yield stress did not change significantly with time during the first 4 h [15]. The tests by the plate device, therefore, were conducted between about 45 min to 3 h after water and cement were in contact. The jar with the cement paste was shaken manually before each subsequent test.

[1] TiO_2 was from Tronox CR-826 (see also footnote 2).

[2] Commercial equipment, instruments, and materials mentioned in this report are identified to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology (NIST), nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

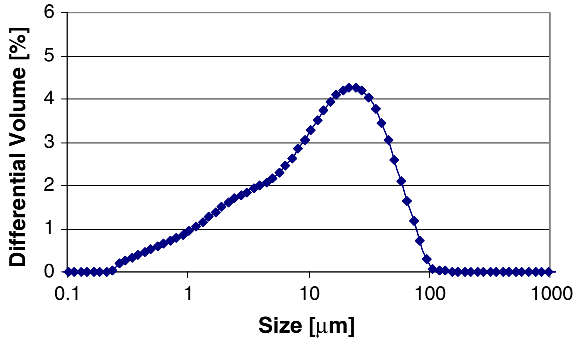


Fig. 7 Cement particle size distribution in isopropanol by laser diffraction method

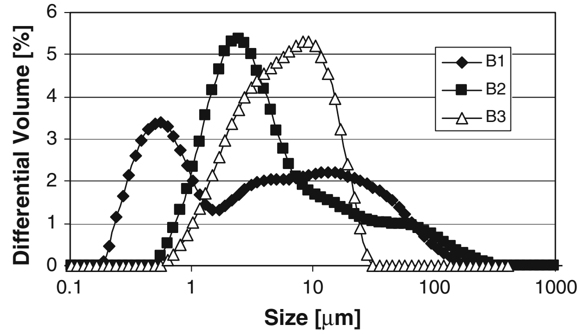


Fig. 8 Bentonite particle size distribution in water by laser diffraction method

4.1.2 Bentonite

Three bentonites of different origins were used: B1 (France), B2 and B3 (USA). Bentonite was selected because it is similar to cement with regards to particle size distribution but it does not react with water and therefore its properties do not evolve with time as in cement paste. Nevertheless, the particles absorb water slowly, which could result in changes in the measured properties over a period of time, but usually measured in months rather than in hours like in cement paste. All three materials were mostly pure commercial bentonites with no additives. The particle size distribution of each bentonite is shown in Fig. 8 as measured in water by laser diffraction. For the measurements in water, bentonite slurry that was over a year old (this would ensure that further water absorption is impossible) was sampled and placed in the laser diffraction device for measurements. It can be seen that the particle size distributions of the three bentonites are quite different. It seems that B1 can disperse better and the particle size is smaller than for B2 and B3. These could partially explain the very different rheological behavior observed for B1 compared to B2 and B3.

The density was tested after the material was dried in an oven at 105°C for 4 h and then kept in a desiccator for 2 days. A Le Chatelier flask with isopropanol was used to measure the particles density. The values obtained were:

- B1: 2,660 ± 20 kg/m^3
- B2: 3,425 ± 20 kg/m^3
- B3: 3,415 ± 20 kg/m^3

The moisture in the bentonite was measured by drying the material at 105°C overnight and was found to be 9.3% for B2 and 7.5% for B3 by mass. The uncertainty is estimated at 0.1%. B1 moisture was not measured.

Bentonite water suspensions B2 and B3 were prepared in the 2 l-plastic containers and the containers were capped and rotated on a 3D mixer[3] intermittently with a roller for at least two months before the suspensions were used for testing. Bentonite B1 was tested after about 1 year. The suspension was mixed on the 3D mixer for 0.5 h before the tests each day, and manually shaken between each test during the day. Bentonite suspensions of B1 and B2/B3 had volume concentrations of 4.0% and 3.1%, respectively (Table 2).

4.1.3 Titanium oxide

The titanium oxide (TiO_2) suspension was prepared from a commercial TiO_2 pigment[1] according to Nguyen et al. [17]. TiO_2 pigment is silica/alumina-treated rutile pigment with pH of 8.0. The solid particles had a specific gravity of 4.0 and contained 93% TiO_2 mass fraction with an average diameter of 0.2 µm. Aqueous 0.01 mol/l KCl solution was used as the dispersing medium to prepare a suspension with a mass concentration of 50% solids (volume concentration of solids = 20%). The suspension was prepared in a 2 l-plastic container and the containers were capped and rotated on a 3D[3] mixer initially for 1 h. Afterwards, the suspension was mixed

[3] A 3D mixer is device that allows a material contained in jar to be tumbled and rolled at the same time. A Turbula was used for this mixing (see also footnote 2).

intermittently by hand for at least 30 s before each tests. The pH of the suspension was adjusted to 7.0 by adding HCl and KOH. All the tests were conducted within 48 h after the TiO_2 suspensions were prepared.

4.2 Other rheometers used

A rheometer that consists of two 35 mm, stainless steel serrated, circular parallel plates was used to determine the yield stress based on a stress growth procedure [16]. The top plate rotates at a controlled shear rate and the torque generated from the resistance of the material tested was measured on the top plate. The bentonite suspensions and cement paste were measured using this system. Due to the difficulty in cleaning the plates, disposable plates of 60 mm in diameter were used to test the TiO_2 suspensions. The temperature was controlled at 23 ± 2°C. The details of the rheometer are described in Amziane and Ferraris [16] and Ferraris et al. [18].

The same rheometer but with a coaxial cylinder configuration was used to determine the yield stress and plastic viscosity of the suspensions based on the Bingham model. The shear rate controlled coaxial cylinder was used. The cylindrical container had an outer diameter of 43 mm. The rotor cylinder had a length (16 mm) and a diameter (22 mm). The rotor had smooth walls as it is customary in a coaxial cylinder rheometer. The torque resulting from the material resistance was measured at the central rotating tool. The shear rate was first changed gradually from 0.5 to 50 s^{-1}, and then decreased gradually to 0.5 s^{-1}.

5 Results and discussion

5.1 Determination of yield stress from the slotted plate device

5.1.1 Interpretation of the measurements

Figure 9 compares typical curves of the force response with time determined from the plate device using the MPS plate (75 mm/no slot and sand blasted) (column a) and typical curves of the shear stress versus shear rate determined from the co-axial rotational rheometer for the suspensions tested (column b).

Practically, it is easier to determine the maximum value than the point where the force response–time curve departs from linearity, particularly for the cement paste and TiO_2 suspension. For these two suspensions, the force–time curves were not as smooth as those of the bentonite suspensions, which made the determination of the points where the force–time curves departed from linearity more difficult. Thus, the yield stress calculated from the force at Point B (maximum) would have less uncertainty than that calculated from Point A, and was therefore used in this work. The decision to use Point B instead of the Point A (representing the yield stress independent from shear rate) is acceptable here because the comparison is between results obtained using the same shear rate. This result can also be compared with the rotational rheometer since the yield stress is recorded at the onset of viscous flow.

From Fig. 9, it appears that the flow (measured using the rotational rheometer) of the bentonite suspensions B2 and B3 may be approximated by the Bingham model (linear), whereas that of the cement paste, bentonite B1, and TiO_2 suspensions may be approximated by the Hershel-Buckley model (not linear and requires three fit parameters).

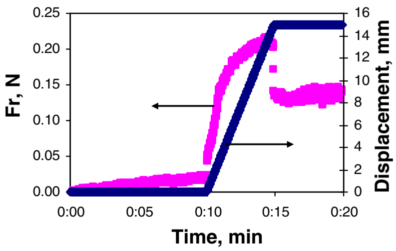
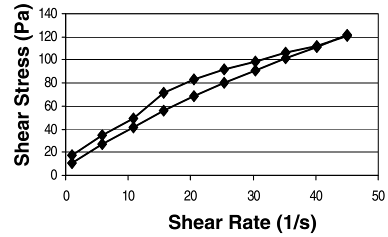
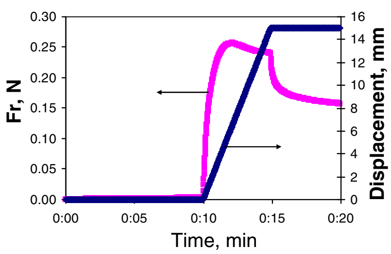
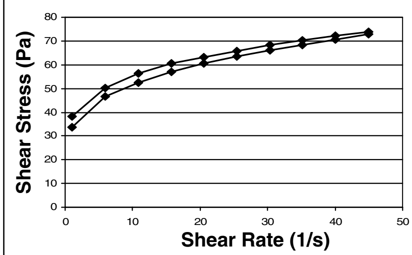
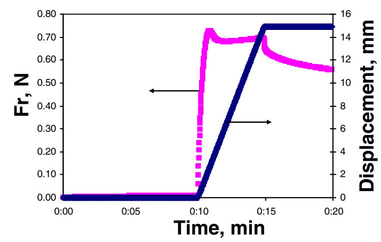
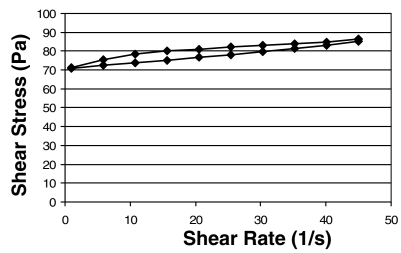
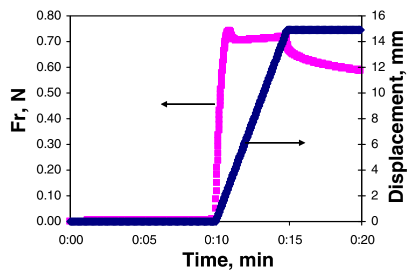
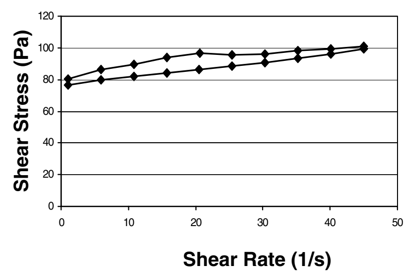
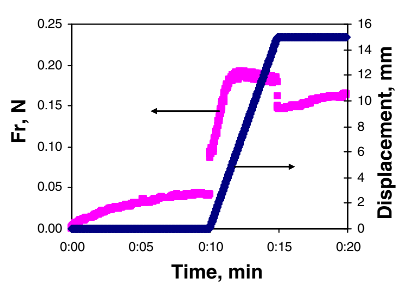
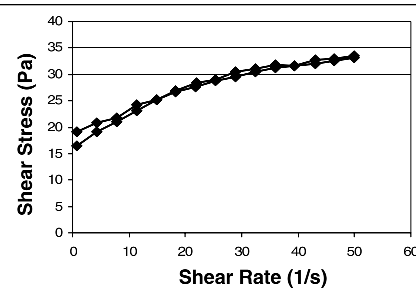
The uncertainty of the results is estimated to be about 10% for the measurements with the rotational rheometer [18, 19]. For the tests using the plate device, the estimation of the uncertainty was harder due to the limited number of tests performed with this device.

5.1.2 Edge effect correction

According to numerical simulations of stress distributions along the plates of different lengths for testing bentonites B1 and B3 [15], it was concluded that:

- for the Bingham type fluids, shorter length plates can be used and the end effect can be ignored, and
- for the Hershel-Bulkley type fluids, a correction factor for the end effect has to be computed in order to obtain accurate yield stress values.

The mean shear stress for the slotted and non-slotted plates was calculated with and without the consideration of an edge-effect correction, and the comparisons are presented in Tables 3 and 4 for the sand blasted and non-sand blasted plates, respectively.

	Typical force response with time determined from the plate device using the MPS plate	Typical curve of shear stress vs. shear rate determined from the co-axial rotational rheometer
Cement paste M		
Bentonite suspension B1		
Bentonite suspension B2		
Bentonite suspension B3		
TiO_2 suspension (A parallel plate was used instead of co-axial rotational rheometer)		
	(a)	(b)

◀**Fig. 9** Comparison of typical curves of the force response with time determined from the plate device using the MPS plate (**a**) and those of the shear stress versus shear rate determined from the co-axial cylinder rheometer (**b**). The data are from one measurements but the uncertainty is estimated to be 10%

From Fig. 9 and the type of model needed to fit the shear stress-shear rate, it would appear that cement paste and bentonite B2 and B3 have no end effects, while the bentonite B1 and TiO_2 should have a correction factor due to end effects. In Tables 3 and 4, the mean stress for all materials is shown. The percentage differences between the values without edge correction and with edge corrections are calculated in relation to the values with an edge effect correction. From the results shown in Tables 3 and 4, it seems that for the B2 and B3 bentonite suspensions, consideration of the edge-effect correction in the calculation of the mean shear stress τ resulted in differences of less than 16% compared with that without the edge-effect correction. For the bentonite B1, the differences were over 20%. The trend seems to be in agreement with the numerical simulation. For the TiO_2 suspension, the difference was relatively small (9%) when the sand blasted plate was used (Table 3), but substantial (about 80%) when a smooth plate was used (Table 4). Thus no clear trend was observed. For the cement paste, however, the difference in the shear stress calculated with or without consideration of the edge correction was substantial, in contradiction with the numerical simulation. The authors have no explanation at this time for the reason of the discrepancy.

For further comparison, therefore, the shear stress with the consideration of edge correction was used. Discussion of these results is presented in Sect. 5.1.3.

5.1.3 *Comparison of yield stress from the slotted and non-slotted plates*

Tables 3 and 4 show that, for the bentonite and TiO_2 suspensions, the mean shear stress τ determined from

Table 3 Mean shear stress τ comparison (sand blasted plates)

Plate type	With edge effect correction τ (Pa)		Without edge effect correction τ (Pa)		% Difference in τ with or without edge correction	
	MPS $\beta = 0$	SL11 $\beta = 0.358$	MPS $\beta = 0$	SL11 $\beta = 0.358$	MPS $\beta = 0$	SL11 $\beta = 0.358$
Cement paste	14.6	30.6	29.2	45.1	100	47
Bentonite B1	25.7	24.2	32.0	30.5	25	26
Bentonite B2	80.5	74.0	88.9	82.3	10	11
Bentonite B3	84.8	80.0	97.2	92.4	15	16
TiO_2 suspension	21.5	21.0	23.4	22.9	9	9

The uncertainty is estimated at 10%

Table 4 Mean shear stress τ comparison (smooth, non-sand blasted plates)

Plate type	With edge-effect correction τ (Pa)		Without edge-effect correction τ (Pa)		% Difference on τ with or without edge correction	
	MP $\beta = 0$	SL14 $\beta = 0.455$	MP $\beta = 0$	SL14 $\beta = 0.455$	MP $\beta = 0$	SL14 $\beta = 0.455$
Cement paste	5.8	23.8	26.7	44.7	360	88
Bentonite B1	26.3	25.1	31.5	30.2	20	20
Bentonite B2	71.5	73.1	81.4	83.0	14	14
Bentonite B3	70.5	77.9	81.8	89.2	16	15
TiO_2 suspension	11.4	11.5	20.6	20.7	81	80

The uncertainty is estimated at 10%

the slotted and solid plates was not significantly different. It was also observed in the experiments that for these suspensions, when the plates (both sand-blasted and smooth) were pulled from the suspensions, the maximum shear strain was probably not through the boundary between the plate surfaces and the suspensions; thin layers of the suspensions were adhering to the plates (so there was almost no slippage). This non-slippage of bentonite on steel surfaces was also observed by Mannheimer [20]. Figure 10 shows an example for bentonite suspension B1. This indicates that the shear was not through the boundary between the plate surface and the suspension, but through the bentonite suspension in a thin layer away from the plate. The calculation of yield stress using the plate device relies on Eq. 3, which considers that τ_S and τ_B as two independent entities. If there is no slippage and τ_S and τ_B are identical, the yield stress cannot be calculated using Eq. 3 [3]. The results obtained for these suspensions are not consistent with those reported by Zhu et al. [3]. They reported (see Sect. 2) that the yield stress increased with β, and with no slippage the stress measured was independent of β.

For the cement paste, the shear stress from the solid (non-slotted) plate was lower than that for the slotted plate, and the slippage appears to be between the plate and the cement paste. This is consistent with the results of Zhu et al. [3].

Fig. 10 Picture showing the bentonite suspension B1 on the smooth medium plate SL11 when the plate was pulled out of the suspension

Because of the above observed differences between suspensions, the yield stress of the cement paste and the other suspensions was calculated via different approaches. For the cement paste, the yield stress τ_B was calculated according to Eq. 4. For the other suspensions, the yield stress was considered as the mean yield stress exerted over the plate surface {i.e. $\tau_B = \tau$ was calculated according to Eq. 1 [$\tau = (F_r - F_D)/2WL$] using the test results from the MPS and MP plates since the shear forces were through the suspensions}.

5.2 Comparison of yield stress of the materials determined by different rheometers and methods

The yield stress of the suspensions determined by different rheometers and procedures are summarized in Table 5.

Yield stress measurements are affected by the time scale of the experiment [5]. The longer the measurement time (the lower the shear rate), the smaller the yield stress value. A stress growth mode was used for the tests with the parallel-plate rheometer and the plate device. Comparing the results from these two tests, the yield stress is reached in a few seconds while using a parallel plate test, and in approximately 100 s in a plate test. The values obtained for the cement paste and bentonite and TiO_2 suspensions are thus lower with the plate tests than those by the parallel-plate rheometer. Another factor that might have contributed to differences between the yield stress determined by the parallel plate rheometer and by the plate device was the surface texture of the plates. For the parallel-plate rheometer, serrated plates were used for the tests except for the TiO_2 suspension. Sand blasted and smooth plates were used with the plate device.

Comparing the results from the test by the co-axial rotational rheometer and the parallel-plate rheometer (Table 5) it is seen that the parallel plate results are almost always higher than those of the co-axial cylinder rheometer. One exception is the bentonite B1 suspension. These discrepancies can be explained by the difference in the type of measurements. The yield stress by parallel plate was determined by a stress growth method, implying that the structure of the material was not disturbed prior to the test. On the other hand, the co-axial cylinder measurements were performed using a Bingham model. The Bingham

Table 5 Comparison of yield stresses determined by the slotted plate device and other rheometers

	Yield stress (Pa)			
	Rheometer		Plate device	
	Parallel-plate configuration (stress growth, shear rate 0.1 s^{-1})	Co-axial cylinder configuration (Bingham, shear rate 50–0.5 s^{-1})	Sand blasted plates (stress growth, platform/plate speed: 0.05 mm/s)	Smooth plates (stress growth, platform/plate speed: 0.05 mm/s)
Cement paste	108	13.2	59.2	45.3
Bentonite suspension B1	40.9	40.8	25.7	26.3
Bentonite suspension B2	93.2	70.2	80.5	71.5
Bentonite suspension B3	119.0	77.5	84.8	70.5
TiO_2 suspension	26.7[a]	21.6[b]	21.5	11.4

The uncertainty is estimated at 10%

[a] Tests were done using a pair of disposable parallel plates without serration due to the difficulty in cleaning the serrated plates

[b] Tests were done using a pair of disposable parallel plates instead of co-axial cylinders

yield stress involves an extrapolation to zero shear rate while the measurements are done from 50 to 0.5 s^{-1}. The structure of the material is destroyed during the high shear portion of the test and therefore the yield stress obtained will be lower. Another factor that might have contributed to the difference for the yield stress determined via the parallel-plate and coaxial cylinder rheometers was the surface texture of the plates and cylinder rotor. Parallel plates had serrated surfaces, whereas the cylinder rotor and inner wall of the outer cylinder were smooth in the coaxial device.

As mentioned earlier, the MPS and SL11 plates were sand blasted, whereas the MP and SL14 plates were smooth and without surface treatment. Table 5 shows that the yield stress of the suspensions determined by the sand blasted plates was higher than that determined by the smooth surface plates except for bentonite suspension B1, which showed similar values of yield stress between the two kinds of plate surfaces. This suggests that the surface texture of the plates does affect the yield stress measurement of the suspensions as in other types of rheometers. This finding seems to be consistent with that reported by Zhu et al. [3].

5.3 Other observations from the tests using the plate device

5.3.1 Force measured without plate movement

Figure 9a shows that for the cement paste and the TiO_2 suspensions, the net force F_r increased with time in the first 10 min before the plate started to move in the suspensions. This phenomenon was not observed for the bentonite suspensions. Table 6 shows the yield stress and plastic viscosity of the suspensions determined by the co-axial rotational rheometer in relation to the volume concentration of the suspensions. The bentonite suspensions had much lower volume concentration than the cement paste and the TiO_2 suspension. For a given type of suspension, the increase in the solid volume generally leads to an increase in the viscosity of the suspension (TiO_2 was measured with a smooth surface and could have lead to a lower or viscosity than expected).

The bentonites are impure clays consisting mostly of montmorillonites. Most clay minerals consist of layers that are stacked parallel to each other. In the suspensions, the bentonite particles absorb water,

Table 6 Yield stress and plastic viscosity of the suspensions determined by the co-axial rotational rheometer

	Volume concentration (%)	Yield stress (Pa)	Plastic viscosity (Pa s)
Cement paste	48	13.2	2.4
Bentonite suspension B1	3.9	40.8	0.8
Bentonite suspension B2	3.1	70.2	0.3
Bentonite suspension B3	3.1	77.5	0.5
TiO_2 suspension	20	21.6[a]	0.2[a]

The uncertainty is estimated at 10%

[a] Tests were done using a pair of disposable parallel plates

possibly several times their original dry mass [21], between the layers containing SiO_2 and Al_2O_3. This results in expansion and swelling of the particles, increase in the volume concentration, and reduction in the inter-particle distance and particle density. The suspensions may behave as gels or "concentrated" suspensions so that either there is no settling of the swollen bentonite particles in the gels or the settling of the particles is limited and unnoticeable in the concentrated suspensions in the first 10 min. The cement paste had limited hydration at short times, particularly when the retarder admixture was added; the paste behaves as a suspension. Due to the difference of specific gravity between cement and water, the cement particles might have settled in the first 10 min even before the plate moved through the paste. The settlement of cement particles may be the reason of the increase in the net force F_r with time registered in the first 10 min. This may also be the cause of the same phenomenon observed with the TiO_2 suspension. An increased sedimentation implies a downward movement of the particles against the plate. In this movement, the plate is pulled down and thus an increase of the force F_r is measured.

A further observation for materials with a stress increase during the first 10 min is that there is also an increase during the relaxation period (following Point C in Fig. 4). A possible explanation is that a small vertical deformation could generate a shear stress at the plate surface, leading to a mass increase in the balance. The mass increase can be due to a vertical consolidation and/or a vertical deformation under the material's own weight. In cement paste, the origin of this deformation was explained by Ovarlez and Roussel [22] by the small settling that can always be measured at the surface of any freshly cast concrete. If cement paste at rest (below the yield stress) behaves as an elastic solid, a deformation (shear strain) must be the origin of the shear stress that is exerted on the plate by the cement paste. The shear strain for cement paste that would induce the paste to yield (critical strain) is relatively low, about 0.0005 [23]. Even a small deformation could generate a large part of the yield stress, since until the critical strain, the stress increases linearly due to the elastic behavior of the material at rest. Tchamba et al. [24] observed that the shear stress at the surface of a static plate increases with time, (i.e. the balance records an increase in mass with time). This shear stress could reach, in specific cases, the yield stress value using a static plate simply immersed into cement paste, without any motion.

In bentonite the situation is different as no increase of the stress is detected in the first 10 min (Fig. 4). This could be due to the fact that bentonite has a critical strain of the order of 0.1 and therefore a higher deformation is needed to overcome the yield stress. As no increase is detected, this would imply that the particles are not moving significantly in the time frame of the experiment (10 min). Bentonite does not sediment as easily as cement paste at the concentration used, leading to no strain and therefore no stress on the plate at rest. In other words, the strain rate generated at the interface between the plate and the material is small and always lower than the critical strain rate. The shear stress on the plate has an unknown value between zero and the yield stress and cannot be detected on the balance, thus the shape of the curve seen in Fig. 4.

5.3.2 *Experimental issues with concentrated suspensions*

One of the difficulties using the plate device for concentrated suspensions is to ensure that the plate is in a vertical position. This is particularly difficult in the case of cement pastes. If the plate is not in a vertical position, it will cause errors because the force measured would be higher than for a vertical plate due to other factors than yield stress.

Another difficulty in determining the yield stress by the plate device resides with the experimental time. Since the shear stress τ_S is associated with the suspension to be tested, the determination of the edge effect correction is essential for suspensions such as cement pastes. This means that the correction factors have to be determined for cement pastes with different w/c ratios or with different types or dosages of admixtures. In order to determine this correction factor, time consuming tests using small, medium, and large plates are required. Cement experiences hydration when in contact with water and the yield stress of the cement pastes increases with time after an initial dormant period. This makes accurate yield stress determination difficult. In this report, a retarder was added to allow the cement paste to be stable for 4 h but the test should be designed for a wide range of cement paste compositions and not only cement paste using a retarder.

6 Summary

The determination of the true yield stress is essential in the description of the flow of suspensions such as cement pastes. A simple rheometer was designed for measuring the yield stress in concentrated suspensions. The scope of this study was to assess whether this newly developed slotted plate device would be usable for dense suspensions such as cement pastes.

A slotted plate device similar to the one used by Zhu et al. [3] and Zhu and De Kee [4] was built. The device was modified to accommodate highly concentrated suspensions with larger particles than used by Zhu et al. [3]. Cement paste, bentonite and TiO_2 suspensions were measured. Traditional rotational rheometers were used for comparison.

Numerical simulations of the plate device [15] show that the end effects (drag force on the upper and lower edges of a plate moving in the suspension) depended on the type of material being measured. For the Bingham type fluids, shorter length plates can be used and the end effect can be ignored. For the Hershel-Bulkley type fluids, a correction factor has to be computed in order to obtain accurate yield stress values.

Experimental results show that the flow (measured using the rotational rheometer) of the bentonite suspensions B2 and B3 may be approximated by the Bingham model, whereas that of the cement paste, bentonite B1, and TiO_2 suspensions may be approximated by the Hershel-Buckley model. For the bentonite suspensions, experimental results confirmed the numerical simulations in terms of the end effect correction for the calculation of yield stress value. For the TiO_2 suspension, no clear trend was observed. For the cement paste, however, the difference between the shear stress calculated with or without consideration of the edge correction was substantial.

Since the yield stress measurements are affected by the time scales of the experiments, the yield stresses of the suspensions determined by the plate device were generally lower than those determined by the parallel-plate rheometer with an exception. The surface texture of the plates affects the yield stress measurement of the suspensions as in other types of rheometers.

It appears that the pattern of stress growth curve and method of yield stress calculation in plate device experiments are affected by the suspension type. Further developments are needed, both in experimental work and in simulation, to adopt this method for concentrated suspensions such as cement pastes.

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References

1. Hackley VA, Ferraris CF (2001) The use of nomenclature in dispersion science and technology. NIST Recommended Practice Guide SP-960-3
2. ACI 238-1R (2007) Report on measurements of workability and rheology of fresh concrete. American Concrete Institute
3. Zhu L, Sun N, Papadopoulos K, De Kee D (2001) A slotted plate device for measurement static yield stress. J Rheol 45(5):1105–1122
4. Zhu H, De Kee D (2006) Slotted-plate device to measure yield behavior of powders; FEM analysis. Ind Eng Chem Res 45:5375–5383
5. Nguyen QD, Boger DV (1992) Measuring the flow properties of yield stress fluids. Annu Rev Fluid Mech 24: 47–88
6. Zhu L, Papadopoulos K, De Kee D (2002) Yield stress measurement of silicon nitride suspensions. Can J Chem Eng 80:1175–1180
7. Picandet V, Ferraris CF, De Kee D (2007) Novel rheometer to measure yield stress of suspensions. In: 5th international RILEM symposium on self-compacting concrete, Ghent, Belgium, pp 335–340
8. Papenhuijzen JMP (1972) The role of particle interactions in the rheology of dispersed systems. Rheol Acta 11:73–88
9. Pryce-Jones J (1952) Studies in thixotropy. Kolloid-Z 129:96–122
10. Van den Tempel M (1971) Structure of multiphase systems. In: Houwink R, de Decker HK (eds) Elasticity, plasticity and structure of matter. Cambridge University Press, Cambridge, pp 123–137
11. Lin SF, Brodkey RS (1985) Rheological properties of slurry fuels. J Rheol 29:147–175
12. Mewis J, Meire C (1984) Yielding in weakly flocculated systems. In: Mena B, Garcia-Rejon A, Rangel-Nagaile C (eds) Advances in rheology 2. Univ Nac Auton Mexico, Mexico City, pp 591–598
13. Nagase Y, Okada K (1986) Heterogeneous behavior after yielding of solid suspensions. J Rheol 30:1123–1142
14. Lombardi G (1985) The role of cohesion in cement grouting of rock. In: Proceedings of 15th international congress on large dam, Lausanne, Switzerland, pp 235–261

15. Ferraris CF, Zhang MH, Zhu H, Picandet V, Peltz MA, Stutzman P, Arnaout C, De Kee D (2008) Measurement of yield stress for concentrated suspensions using a plate device, NIST Technical Note 1494. National Institute of Standards and Technology, US Department of Commerce
16. Amziane S, Ferraris CF (2007) Cementitious paste setting using rheological and pressure measurements. ACI Mater J 104(2):137–145
17. Nguyen QD, Akroyd T, De Kee D, Zhu L (2006) Yield stress measurement in suspensions: an inter-laboratory study. Korea Aust Rheol J 80(1):15–24
18. Ferraris CF, Obla K, Hill R (2001) The influence of mineral admixtures on the rheology of cement paste and concrete. Cem Concr Res 31(2):245–255
19. Ferraris CF (2006) Concrete rheology: knowledge and challenges. In: 2nd international RILEM symposium on advances in concrete through science and engineering, Quebec, Canada
20. Mannheimer RJ (1990) Laminar and turbulent flow of cement slurries in large diameter pipe—a comparison with laboratory viscometers. In: Proceedings of international conference organised by the British Society of Rheology—rheology of fresh cement and concrete, Liverpool, UK, pp 147–158
21. Luckham PF, Rossi S (1999) The colloidal and rheological properties of bentonite suspensions. Adv Colloid Interface Sci 82:43–92
22. Ovarlez G, Roussel N (2006) A physical model for the prediction of lateral stress exerted by self-compacting concrete on formwork. Mater Struct 39(2):239–248
23. Struble LJ, Schultz MA (1993) Using creep and recovery to study flow behaviour of fresh cement paste. Cem Concr Res 23:1360–1379
24. Tchamba JC, Amziane S, Ovarlez G, Roussel N (2008) Lateral stress exerted by fresh cement paste on formwork: laboratory experiments. Cem Concr Res 38:459–466